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ISOTHERMAL AGING OF IM7/8320 AND IM7/5260

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ABSTRACT

Isothermal aging was conducted on two composite systems being considered as possible candidates for the next generation supersonic transport. The composite systems were IM7/5260, carbon/thermoset (toughened bismaleimide) and IM7/8320, carbon/amorphous thermoplastic. The materials were isothermally aged for a total of 5000 hours at 125°C and 175°C. These temperatures are approximately equivalent to the upper skin temperatures of an aircraft flying at Mach 2.0 and Mach 2.4 flight, respectively. The variations of the following properties were determined as a function of aging time: weight loss, moduli, glass transition temperature, microcracking, and modulus and strength of a ± 45 laminate. The difficulties and accuracy of strain measurements are also discussed.

INTRODUCTION

For the new generation of supersonic civilian transport aircraft to be economically viable, they must meet certain size and weight criteria. By constructing the airframe from polymeric composites these criteria may be met. A viable supersonic transport aircraft is anticipated to require a useful life of 60,000 hours, 75 percent of which will be at supersonic speeds and hence high temperature. Thus, the airframe material must be able to maintain its structural properties at that temperature for that length of time. Indeed, the materials may need to be certified for twice the anticipated flight time; therefore, screening the materials for durability becomes an important issue. One type of screening is related to a material's resin glass transition temperature, T_g . If a material has a T_g higher than the use temperature then it is a possible candidate for use on a supersonic transport aircraft. However, the T_g

is not an indicator of the material's thermo-oxidative stability (TOS), or its ability to maintain its material properties for long durations at high temperature. A material's TOS can be partly investigated by conducting short term isothermal aging tests and monitoring how various material properties vary with aging time. If the material does not retain the majority of its baseline material properties over a short period of time, then it cannot be used for the application for which it was tested.

This paper reports the results of a short term aging program. Two materials, a carbon/amorphous thermoplastic and a carbon/thermoset (toughened bismaleimide) with high T_g s were chosen as model materials. Specimens to determine various properties were manufactured and were exposed to elevated temperatures in a mechanically unloaded state for times up to 5000 hours. The two aging temperatures used were 125°C and 175°C. These temperatures are the approximate skin temperatures at Mach 2.0 and Mach 2.4 flight, respectively. Specimens to determine the composite longitudinal, transverse and shear moduli, E_{11} , E_{22} , and G_{12} , respectively, were manufactured to determine how these moduli may vary with time at temperature. These data may be used in damage tolerance modeling such as that described in Ref. 1. Matrix dominated ± 45 specimens were also manufactured to determine how the strength and modulus of a ± 45 laminate vary with time at temperature. Other specimens to determine changes in microcracking, T_g , and infrared spectra were also manufactured. In addition, to assess the accuracy of strain measurements for long-duration tests (e.g. creep), a study was conducted on the effects of temperature on the stability of the strain gauge and test material. It was

expected that the results from this study would aid in the selection of strain gauges for long term tests and lend insights into how the aging process of the test materials may affect the measured strain.

MATERIALS

The composite materials used throughout this study were IM7/5260, a carbon/BASF thermoset (toughened bismaleimide) and IM7/8320, a carbon/Amoco amorphous thermoplastic (polysulfone). The fiber, IM7, was an intermediate modulus carbon fiber manufactured by Hercules. Composite panels were manufactured with the lay-up required for the specimens described in the Specimen Configuration subsection and the specimens were cut from these panels. The IM7/5260 composite panels were cured in an autoclave according to the following cycle: The temperature was raised from ambient (nominally 23°C) to 150°C at 1.7°C per minute under vacuum. The temperature was held at 150°C for 30 minutes and then the pressure was increased to 0.59 MPa. The temperature was then raised to 192°C at 1.7°C per minute and held for 4 hours. Following the cure, the panels were postcured at atmospheric pressure with the following cycle: The temperature was increased from ambient to 220°C at 2.8°C per minute and held for 6 hours. The oven was then switched off and the panels were allowed to cool overnight in the oven (typically 12-15 hours). The IM7/8320 panels were consolidated in a press. The temperature was raised to 350°C at approximately 12°C per minute. The pressure was increased to 5.5 MPa and held for 5 minutes. The temperature was then decreased at approximately 10°C per minute and the pressure released when the panel reached

room temperature. Following postcure or consolidation all the panels were C-scanned to determine panel quality. A steel specimen was also machined for the strain measurement study, as detailed later.

Specimen Configuration

Specimens were manufactured to determine various material properties. The specimen lay-ups, dimensions, number of replicates per data point, and fiber volume fractions, V_f , are given in Table 1. To determine the moduli, and the effect of accuracy of strain measurements, high temperature strain gauges were bonded to selected specimens. The gauges used were Micro-Measurements WK-00-250BG-350 gauge with M-Bond 600 gauge adhesive and 220°C solder. This combination of gauge and adhesive seemed to minimize the problems associated with coefficient of thermal expansion mismatch to the composite, and gauge/adhesive creep. The manufacturer's instructions were followed for gauge mounting and installation. The specimens for T_g determination were cut from the end of the $[\pm 45]_2$ specimens. No end tabs were used.

EXPERIMENTAL PROCEDURES

Prior to aging, all the aging study specimens were dried in air at atmospheric pressure with the following cycle: 1 hour at 95°C, 1 hour at 110°C, 16 hours at 125°C and 1 hour at 150°C. The specimens were placed immediately on metallic wire racks inside isothermal aging ovens. Some specimens were weighed before placing in the ovens, as described below. Forced convection horizontal airflow was used in two ovens, one oven at 125°C and the other at 175°C. The average air velocity was

approximately 0.75 m/sec. The specimens were exposed to the aging temperature for times up to 5000 hours. Before removal of the specimens, at the specified time, the ovens were switched off and allowed to cool down for 2 to 3 hours. The heating and cooling in the oven were not accounted for because the specimens undergoing 5000 hours of exposure only saw 5 thermal cycles, which were thought to have negligible effect.

The quantity of microcracking was determined using an optical microscope with magnifications of 400X. To determine weight loss, the $[\pm 45]_2$ specimens were weighed at room temperature after having been dried, but before being aged and then again after each aging interval. Three specimens were weighed and an average weight loss determined. An electronic balance with a weight measurement precision of 0.001g was used. The weight loss was calculated as a percentage from:

$$\frac{W_{original} - W_{aged}}{W_{original}} \times 100 \quad (1)$$

where W is weight.

To determine the T_g , small pieces were cut from the $[\pm 45]_2$ specimens. Glass transition temperatures were determined for IM7/8320 samples using a differential scanning calorimeter (DSC). Experiments were conducted in air at a heating rate of 20°C per minute. The T_g s of the IM7/5260 specimens were not definable using the DSC because of the lack of definition of the transition point of the trace. Therefore, a thermomechanical analyzer (TMA) was used to measure the T_g s of IM7/5260. A 5 gram load was applied on the penetration probe and the material was heated above

its softening temperature at a rate of 10°C per minute. The temperature at the intersection of the local tangents prior to and after the softening point was considered to be the T_g . To ensure compatibility of results the TMA was also used on the IM7/8320 specimens and the T_g values compared with those from the DSC. The T_g s between the two methods were similar.

Diffuse reflectance Fourier transform infrared spectroscopy (DRFTIR) was also carried out to inspect for signal variations associated with chemical changes induced by thermal aging. Fourier transform infrared spectra were obtained for controls (zero aging time) and compared with spectra of samples aged for various lengths of time.

The tests to determine the aging induced changes in material moduli, E_{11} , E_{22} , and G_{12} , were conducted in a screw driven machine at ambient temperature. Separate longitudinal and transverse gauges were used to determine G_{12} according to the method given in ASTM Standard D3518. Two specimens of each type were used and the results averaged. The specimens were loaded under displacement control at 0.5 mm/minute (approximately 50 $\mu\epsilon$ /sec) to less than 15 percent of their ultimate strength and then unloaded. Stress-strain, σ - ϵ , traces were taken on an X-Y plotter. The specimens were then returned to the aging ovens. The total time the specimens were out of the oven was no more than four hours. These specimens were not used to determine weight loss.

To determine the strength and modulus of the $[\pm 45]_2$ specimens, they were tested in a servo-hydraulic test stand under load control at ambient room temperature. Three specimens per data point were used. To prevent specimen damage from the

gripping abrasive gauze material was placed between the grip faces and the specimens. The specimens were loaded to failure at approximately 1700 N/minute. This is approximately equivalent to $20 \mu\epsilon/\text{sec}$ in the linear range of the $\sigma-\epsilon$ curve. Longitudinal strain was measured using extensometers with a 25mm gauge length attached to the specimen. Load and strain were recorded using a data acquisition system.

Strain Measurement Study

The first step in the strain measurement study was the selection of a high temperature strain gauge that would be suitable for isothermal creep tests over a large range of strains [2,3]. To confirm the selection of the gauges used, the composite and steel specimens were subjected to a 16 hour drying period at 110°C and a two hour postcure cycle at 170°C and then were subjected to 150°C without mechanical load. The strain gauge adhesive postcure temperature was 20°C above the test temperature, as recommended by the manufacturer. The adhesive post cure temperature was also lower than the post cure temperature of the IM7/5260 and the consolidation temperature of the IM7/8320. To help quantify the observed behavior of the IM7/5260 material, discussed in the Results section below, five instrumented, isothermal tests were conducted on separate specimens using different thermal cycles for each specimen. These cycles were broken down into three basic segments:

1. Precondition - A constant temperature segment for 16 hours designed to remove residual moisture,
2. Gauge Postcure - A 2 hour constant temperature segment to postcure the

strain gauge adhesive, followed by a cooling to room temperature.

3. Test - A constant temperature segment lasting for the duration of interest. A comparison of the thermal histories of the five IM7/5260 specimens is given in Table 2. All five tests were conducted with the test segment extending for at least 150 hours. The tests were performed in an air circulating oven attached to a test stand.

RESULTS AND DISCUSSION

The next section presents the results of the various tests conducted with appropriate discussion.

Microcracking

Table 3 documents the onset of microcracking for the two materials at the two different temperatures. Visual observation of the edges of the IM7/5260 specimens showed extensive microcracking at the edges after 500 hours of aging at 175°C. After 500 hours at 175°C the microcracks ran across each ply thickness and were spaced approximately 2 ply thicknesses apart. A photomicrograph of the edge of a $[0/90]_{6s}$ and of a $[90]_{12}$ IM7/5260 specimen that had been aged for 2000 hours at 175°C are shown in Figs. 1a and 1b, respectively, at a magnification of 400X. After 2000 hours the microcracking was very severe, with the microcracks running along the length as well as across the ply thickness throughout the total thickness. The IM7/5260 showed minor cracking in the outer 2 or 3 plies after 5000 hours at 125°C. The IM7/8320 specimens showed only minor microcracking in the outer 2 or 3 plies

after 5000 hours of aging at 175°C and no microcracking after 5000 hours at 125°C. The duration of these tests was only 5000 hours. It is possible that after 60,000 hours microcracks will develop more extensively even at 125°C and long term aging tests should be considered.

Weight Loss

A plot of weight loss versus aging time for both materials at both temperatures is shown in Fig. 2. The weight loss values are an average of three specimens with the worst scatter being 5% of the mean. For both materials aged at 125°C there was no significant weight loss with the weight loss shown in Fig. 2 being within experimental error. Similarly for the IM7/8320 aged at 175°C there was no significant weight loss. However, there was significant weight loss for the IM7/5260 specimens aged at 175°C. After 5000 hours the specimens lost approximately 2.0 percent of their weight, which correlates with the extensive microcracking.

Glass Transition Temperature

The results of the T_g measurements for all specimens are summarized in Fig. 3. The T_g of IM7/5260 aged at 125°C and 175°C did not change until after 5000 hours of aging, at which time the T_g increased approximately 10°C which may have been caused by continued crosslinking which, in turn, may result in embrittlement of the material.

The T_g of IM7/8320 did not change after 5000 hours of aging at 125°C, however, the same material exhibited an increase in T_g after 500 hours of aging at 175°C when T_g was measured using DSC. A spot check using the TMA indicated

that this increase in measured T_g was not duplicated if the TMA was used. The higher T_g was a result of the DSC experiment having been conducted at a heating rate that was faster than the cooling rate that the sample was subjected to when it was aged at 175°C. A combination of the information on T_g 's with the summary of microcracking observations in Table 2 reveals that there does not appear to be a direct relationship between T_g and microcracking.

Infrared Spectroscopy

The infrared spectra of an IM7/5260 specimen, unaged and aged at 175°C for 1000, 2000 and 5000 hours, are shown in Fig. 4. At longer aging times the carbon-hydrogen band at 2974 cm^{-1} wavenumber [4] decreased and the oxygen hydrogen band between 3000 cm^{-1} and 3600 cm^{-1} wavenumber increased. These changes may be associated with continued crosslinking in the resin which would be consistent with the increase in T_g observed at longer aging times. There were no significant chemical changes detected in any of the other specimens studied.

Laminate Modulus and Strength

The variation of E_{11} with time is shown in Fig. 5. Unfortunately, the gauges became disbonded on the IM7/5260 E_{11} specimens aged at 125°C and no data were taken. Much scatter may be observed in the data in Fig. 5. Tests for E_{11} are very sensitive to alignment of the specimen and it is possible that some of this scatter is from misalignment and any possible trend from aging is hidden. Generally, it is expected that E_{11} would vary little with aging time but the results are inconclusive.

Because of microcracking a difficulty arose in determining E_{22} . The σ - ϵ curve

for the same IM7/5260 specimen after 100 and 2000 hours of aging at 175°C are shown in Fig. 6. After 2000 hours of aging the initial portion of the σ - ϵ curves was nonlinear. This nonlinearity was probably caused by microcracks opening or closing directly under, or near, the strain gauge. The cracks would open or close depending on their orientation to the loading, Fig. 1b. Once the crack movement had stabilized, that is they became fully open or closed, the σ - ϵ curve became linear. It was the linear portion that was used to determine the modulus. It is probable that the loading, even at 15% of the ultimate stress, may have caused the microcracks to grow. Hence, it may be more appropriate in future tests to determine E_{22} from a 0/90 laminate, where the 0° plies would restrict the crack movement in the 90° plies, and to back out the value of E_{22} . The use of extensometers, which have a larger gauge length than a strain gauge, may also be required. The variation of E_{22} with time is shown in Fig. 7. The general trend in all cases is for E_{22} to increase with aging time except for the IM7/5260 specimens aged at 175°C which showed a decrease in E_{22} after 5000 hours of aging. The increase in E_{22} with aging is consistent with the change in T_g of IM7/5260 and other findings [5], and the decrease in the one case is consistent with the decrease in modulus observed in the presence of matrix cracks caused by mechanical loads [6].

The variation of G_{12} with aging time is shown in Fig. 8. Similar problems (microcracks opening and closing) to the E_{22} measurements were experienced with the $[\pm 45]_{2s}$ specimens. Also, this lay-up typically has a nonlinear σ - ϵ curve, hence, a sketched tangent through the origin was used to determine the modulus. From Fig.

8, there is little evidence of a change in G_{12} after 5000 hours of aging at either temperature for either material.

The σ - ϵ curves to failure, in the x direction, of IM7/5260 and an IM7/8320 $[\pm 45]_{2s}$ laminates after 2000 hours of aging at 175°C are shown in Fig. 9. The IM7/5260 laminate has an initially linear σ - ϵ curve with a definable maximum load at failure. However, the IM7/8320 has a less linear σ - ϵ curve and the modulus, E_x , was taken from a graphical linear fit to the curve. Also, the IM7/8320 laminate shows no definable maximum. The load kept on increasing as the fibers rotated to be more parallel with the loading direction. Hence, no strength data could be obtained for the IM7/8320 specimens. The variation of E_x for the $[\pm 45]_{2s}$ specimens with aging time is shown in Fig. 10. For both materials and aging temperatures, there was an initial increase in E_x which began to level off after 1000 hours and then began to decrease. Also, shown in Fig. 10 is the laminate modulus calculated from classical lamination theory, CLT, [7] using the material properties at the specific aging times from Figs. 5, 7, and 8. The calculated modulus is similar to those measured experimentally but does not show the same trend of an increase and then a decrease.

The variation in strength of the IM7/5260 $[\pm 45]_{2s}$ specimens is shown in Fig. 11. Each individual data point is an average of three specimens. The strength decreased with time for the specimens aged at 175°C, showing a 35% decrease after 5000 hours. The specimens aged at 125°C exhibited a 15% decrease after 5000 hours.

The microcracking did not greatly alter the material moduli but the strength of

a ± 45 laminate was reduced. It is possible that the strength and toughness of the material will be affected more by aging and microcracking than the moduli [8], and hence strength and toughness characterization are important.

Strain Measurement Study

For the strain measurement study, typical values for the measured free thermal strains at 150°C versus time for the unloaded steel and two composite specimens are shown as numerically smoothed curves in Fig. 12. The strain/time behavior for the steel and IM7/8320 materials indicate very little change in apparent strain for periods of at least 100 hours. However, the IM7/5260 material exhibited an apparent strain that decayed steadily before leveling off after approximately 100 hours. For long term testing, the relative strain stability exhibited by the steel and IM7/8320 is more desirable than the unstable behavior exhibited by the IM7/5260. Results from the IM7/8320 and steel materials indicate that strain gauge and adhesive creep is not the cause of the decaying strain for IM7/5260.

To further investigate the effect observed on the IM7/5260, the material was subjected to five different thermal histories as given in Table 2. An example thermal and strain history is given in Fig. 13. The apparent strain in the test specimen decays during both the preconditioning and test segment, while a relatively stable strain measurement occurs during the gauge post-cure segment. The strain decrease in the preconditioning segment appears to continue for the duration of the segment, while the strain decrease in the test segment appears to reach a limiting value after about 100 hours. The irregularity in the data is from signal noise in the strain gauge

circuitry. The trends given in Fig. 13 occurred for all five of the tests depicted in Table 1. The only significant difference between specimens was the relative initial strain levels from the segment temperatures. The strain shown in Fig. 13 is the free thermal strain or $\alpha_x \Delta T$, where α_x is the coefficient of thermal expansion in the x direction of a $[30]_{12}$ laminate and ΔT is the test temperature minus the stress free temperature. Hence for the free thermal strain to decrease either the stress free temperature of the laminate, or the coefficient of thermal expansion is changing because chemical changes are occurring in the composite.

Aging will occur in most polymers [9] and may be comprised of physical aging, which is reversible, or chemical aging, which is irreversible, or a combination of these processes. Physical aging will be evident in the linear viscoelastic range at temperatures below T_g for IM7/8320 [10]. Although similar physical aging data on the IM7/5260 material is not available, data from this work indicate aging does occur. The results presented in Fig. 13 indicate that the chemical aging process is more significant in the IM7/5260 than the IM7/8320. The ability to rejuvenate or remove any prior physical aging effects in a polymer by taking the material to temperatures above the T_g may prove to be difficult because of the continuing chemical aging process which effectively makes T_g an unknown quantity. However, the aging process in the IM7/5260 does not prevent long duration testing of this material.

In conclusion, several important matters have arisen in this work which will help in developing test methods for long term aging studies. A problem arose in the determination of modulus when the composite is aged to such a degree that matrix

cracks occur. These cracks cause the σ - ϵ curves to become non-linear and can even give false readings of strain if a gauge is located directly over a crack. In addition, when loading the specimen to obtain a modulus value these cracks may grow. A further problem was noted with the gauge adhesive also aging with the composite.

A possible solution to these problems would be to test a 0/90 laminate and to back out the value of E_{22} . Also, the use of extensometers may circumvent both the gauge adhesive failure and the local presence of microcracks because they test a larger gauge length. The implications of the strain measurement study are that aging effects should be quantified and documented to ensure repeatability and accuracy in the experimental data. In addition, gauge type, gauge adhesive, and apparent strain compensation technique should be reported to help ensure that the recorded strains are only from the mechanical strains in the test material. Finally, the implications of the data in this study are that both materials showed some changes in material properties at both aging temperatures after only 5000 hours of aging. Hence, questions must be placed on the viability of either of these materials on a supersonic transport which will fly at Mach 2.0 or higher for 60,000 hours.

SUMMARY

Isothermal aging was conducted on two composite systems being considered as possible candidates for the next generation supersonic transport. The composite systems were IM7/5260 a carbon/thermoset (toughened bismaleimide) and IM7/8320 a carbon/amorphous thermoplastic. The materials were aged for a total of 5000 hours at 125°C and 175°C. The variation of the following properties were determined as a

function of aging time: weight loss, moduli, glass transition temperature, microcracking, and modulus and strength of a ± 45 laminate. At 125°C the IM7/5260 showed minor microcracking and no significant weight loss or change in moduli. At 175°C the IM7/5260 showed weight loss and microcracking after 5000 hours, but showed little change in moduli and a 35 percent reduction in strength after 5000 hours. Glass transition temperature increased significantly for IM7/5260 aged at 175°C. At 125°C the IM7/8320 showed no microcracking and only minor cracking at 175°C and no significant weight loss or change in modulus for either temperature. No changes in T_g 's were detected for either material aged at 125°C. The IM7/5260 ± 45 specimens aged at 125°C showed a 15 percent decrease in strength after 5000 hours. Also, the effect of strain measurements were investigated and it was concluded that any conditioning cycles to prepare the specimens may have an effect on the measured strain and must be documented.

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REFERENCES

1. Martin, R.H., "Delamination Onset in Polymeric Composite Laminates Under Thermal and Mechanical Loads," High Temperature and Environmental Effects on Polymeric Composites, ASTM STP 1174, C.E.Harris and T.S. Gates, Eds., American Society for Testing and Materials, Philadelphia, 1993.

2. Sullivan, J.L., "Measurement of Composite Creep," *Experimental Techniques*, September/October, 1991, pp. 32-37.
3. Tuttle, M.E., "Fundamental Strain-Gage Technology-Section IIIA," Manual on Experimental Methods for Mechanical Testing of Composites, Ed. M.E. Tuttle and R.L. Pendleton, Society for Experimental Mechanics, 1989.
4. Bellamy, L.J., *Infrared Spectra of Complex Molecules*, Vol. 1, 3rd ed., Chapman and Hall, New York, 1975.
5. Beckwith, S.W. and Wallace B.D., "Effects of Aging and Environmental Conditions on Kevlar/Epoxy Composites," SAMPE Quarterly, July 1983, pp. 38-45.
6. Caslini, M., Zanotti, C., and O'Brien T.K., "Study of Matrix Cracking and Delamination in Glass/Epoxy Laminates," *Journal of Composites Technology and Research*, Vol. 9, No. 4, Winter 1987, pp. 121-130.
7. Jones, R.M., *Mechanics of Composite Materials*, McGraw-Hill Book Company, New York, 1975.
8. Street, K.N., Russell, A.J., and Bonsang, F., "Thermal Damage Effects on Delamination Toughness of a Graphite/Epoxy Composite," *Composites Science and Technology*, 32 (1988), pp. 1-14.
9. Struik, L.C.E., *Physical Aging in Amorphous Polymers and Other Materials*, Elsevier Scientific Publishing Company, New York, 1978.
10. Hastie, R.L. and Morris, D.H., "The Effect of Physical Aging on the Creep Response of a Thermoplastic Composite." High Temperature and Environmental Effects on Polymeric Composites, ASTM STP 1174, C.E. Harris and T.S. Gates, Eds., American Society for Testing and Materials, Philadelphia, 1993.

Test	Lay-up	Number of specs. per data point	Dimension (mm)	V _f %	
				IM7/8320	IM7/5260
E ₁₁ , V ₁₂	[0] ₁₂	1	250x25	63.5	56.8
E ₂₂	[90] ₁₂	2	250x25	63.8	56.8
G ₁₂	[±45] _{2s}	2	250x25	62.5	54.6
weight loss	[±45] _{2s}	3	250x25	62.5	54.6
E _x	[±45] _{2s}	3	250x25	62.5	54.6
T ₉	[±45] _{2s}	1	5x5	62.5	54.6
strain gauge study	[30] ₁₂	1	240x25	-	-
micro-cracking	[0/90] _{6s}	1	20x20	62.5	54.6

The fiber volume fraction was calculated from

$$V_f = \frac{FAW N}{10 FD t} \%$$

where FAW = fiber areal weight = 144 g/m² for IM7/8320
= 145 g/m² for IM7/5260
N = number of plies
FD = fiber density = 1.77 g/cm³
t = average specimen thickness

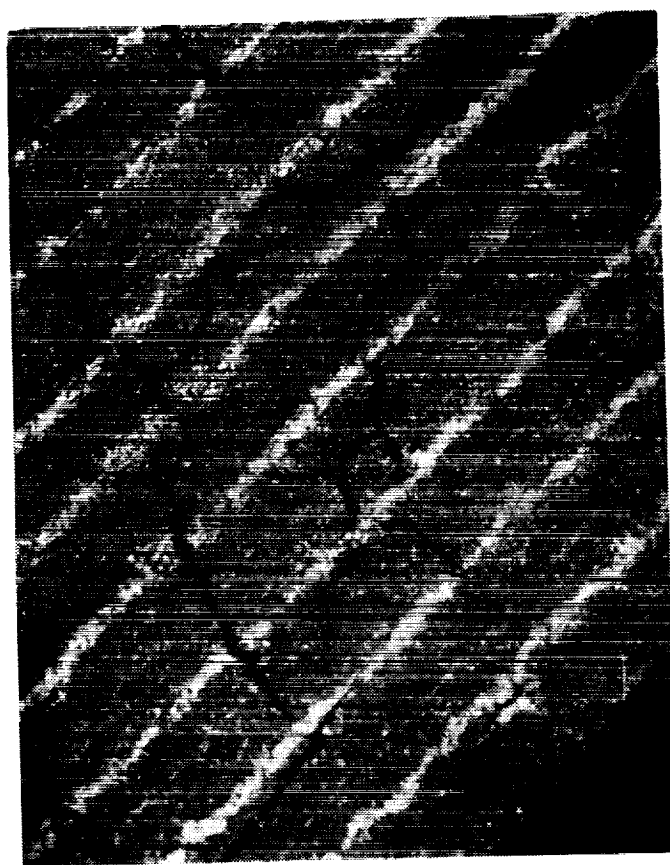
Table 1. Specimen Configurations

Specimen Number	16 hour precondition temperature	2 hour gauge post cure	2 hour cool	> 150 hour test
1	110°C	90°C	23°C	70°C
2	110°C	145°C	23°C	125°C
3	110°C	145°C	23°C	145°C
4	110°C	145°C	23°C	170°C
5	110°C	170°C	23°C	150°C

Table 2 Thermal Histories of IM7/5260 [30]₁₂ specimens

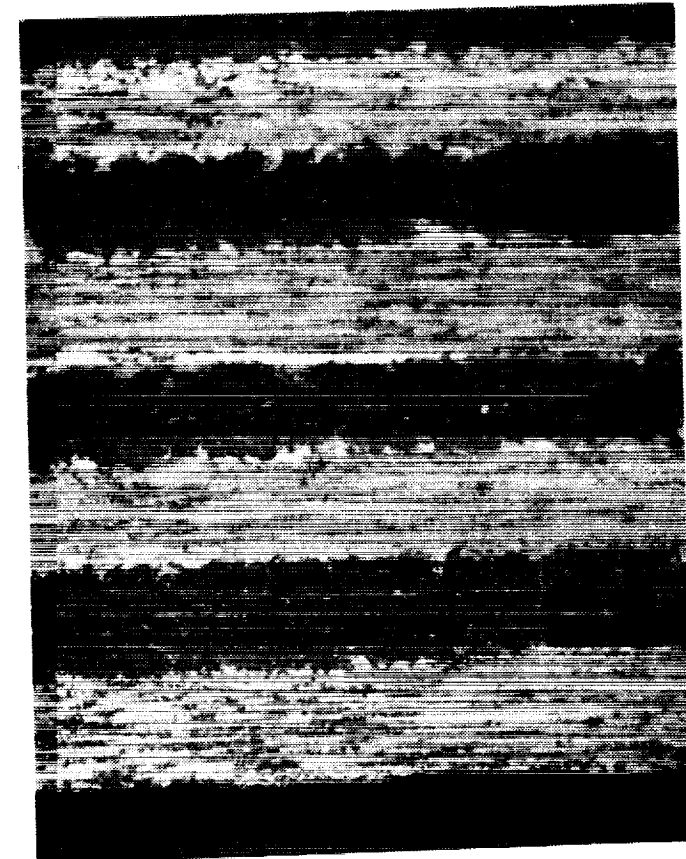
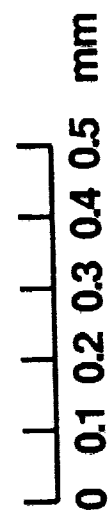
TIME (hours)	IM7/5260		IM7/8320	
	125°C	175°C	125°C	175°C
100	no cracks	no cracks	no cracks	no cracks
200	no cracks	no cracks	no cracks	no cracks
500	no cracks	cracks throughout, 2 ply thicknesses apart	no cracks	no cracks
1000	no cracks	extensive cracks	no cracks	no cracks
2000	minor cracks	extensive cracks	no cracks	no cracks
5000	minor cracks in outer 2-3 plies	extensive cracks	no cracks	minor cracks in outer 2-3 plies

Table 3 History of Microcracking



[0/90]6s

(a)



[90]12

(b)

Fig. 1 Microcracking in IM7/5260 specimens aged at 175°C for 2000 hours

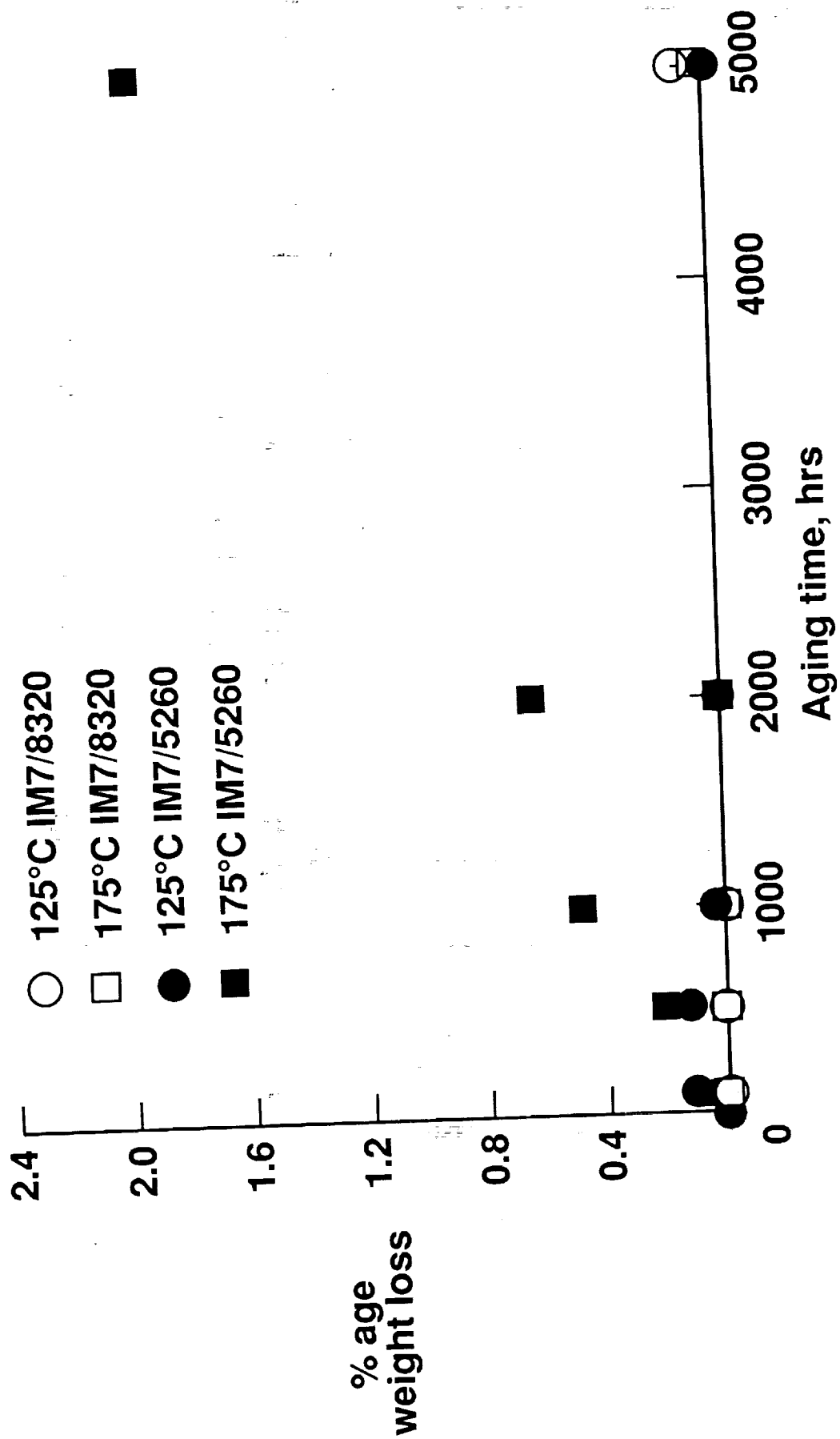


Fig. 2 Weight loss versus aging time

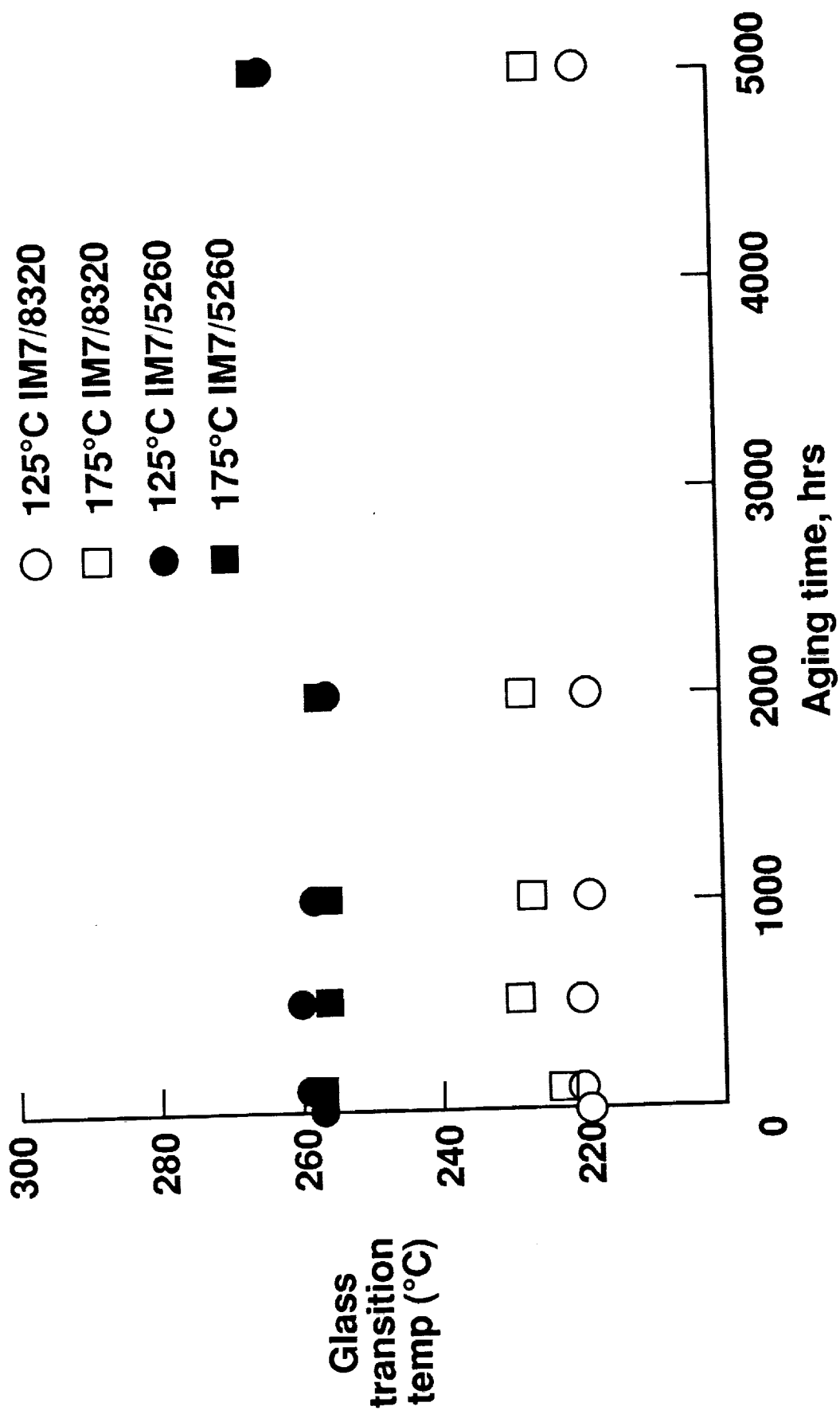


Fig. 3 Glass transition temperature versus aging time

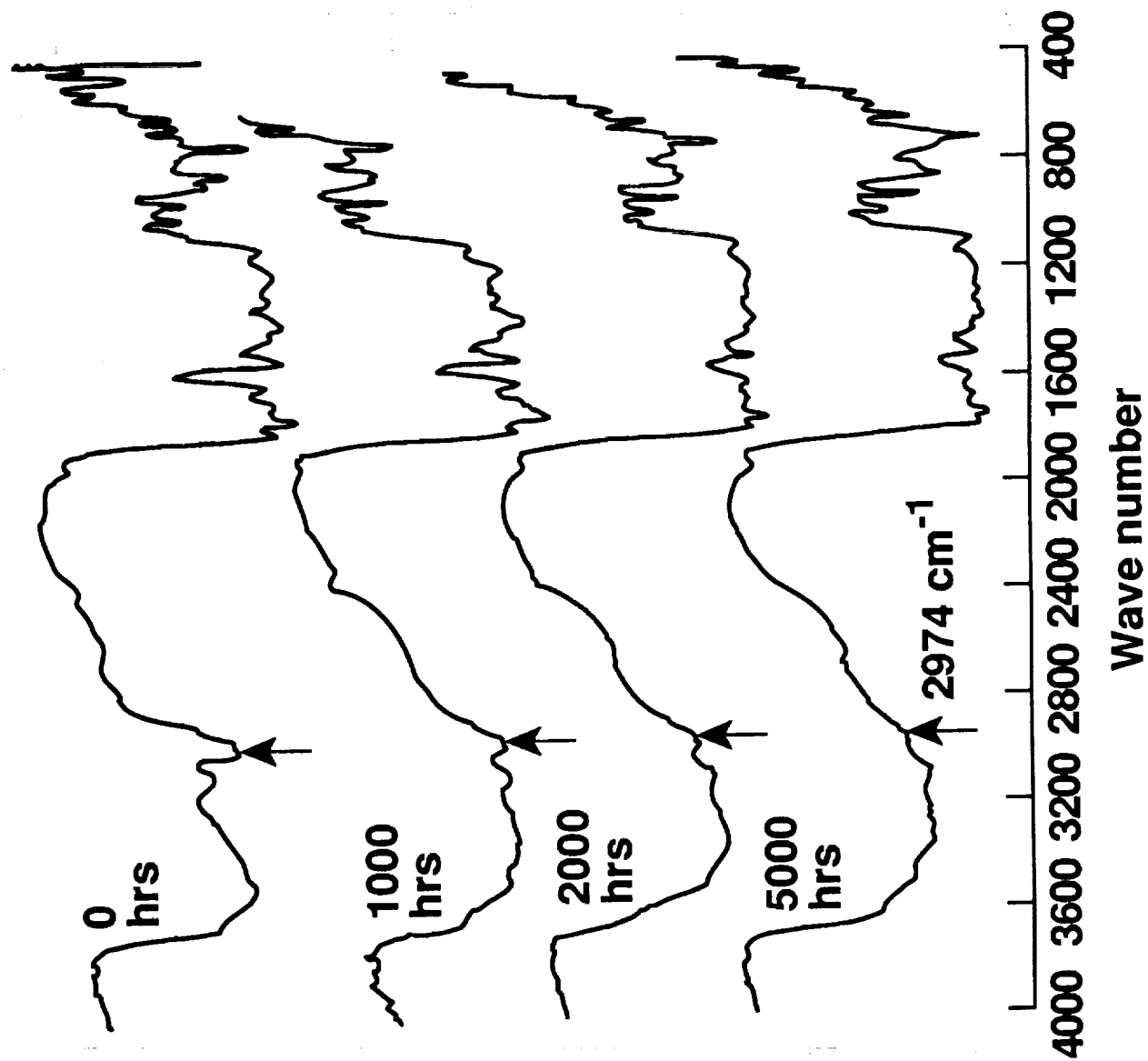


Fig. 4 Infrared spectra for IM7/5260 aged at 175°C for different lengths of time

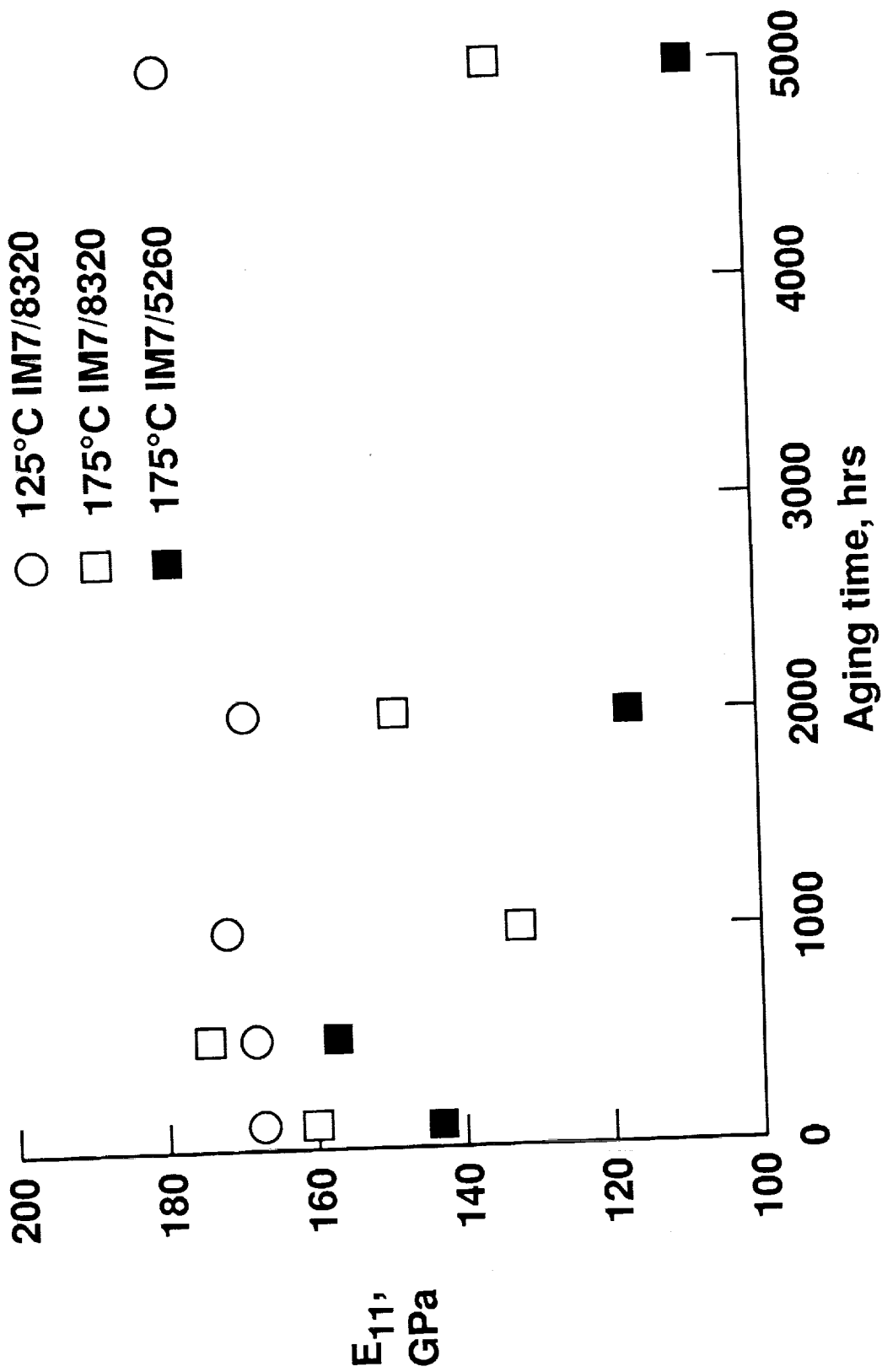


Fig. 5 Longitudinal modulus (E_{11}) versus aging time measured at room temperature

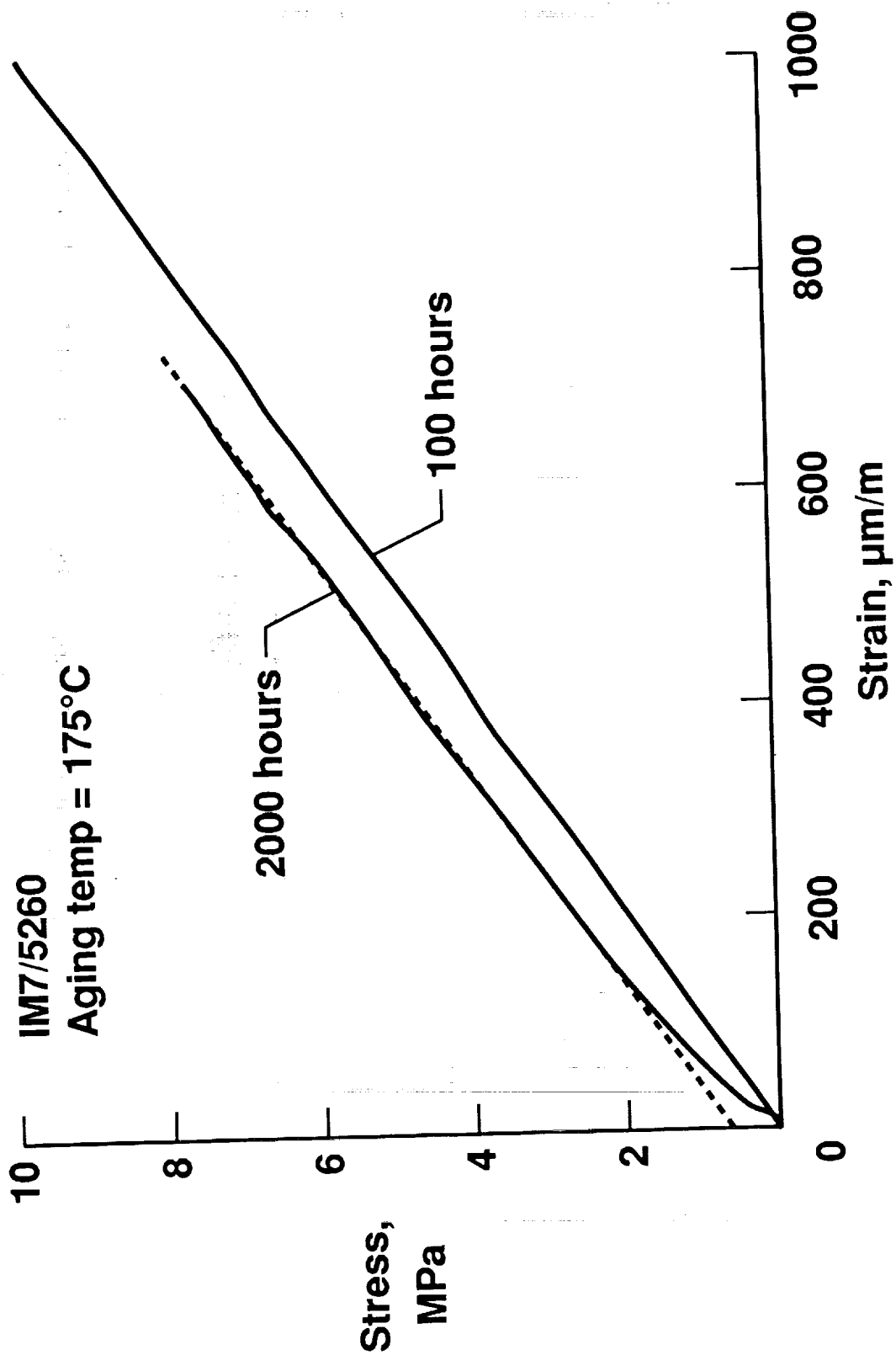


Fig. 6 Stress strain curves for transverse modulus (E_{22}) determination

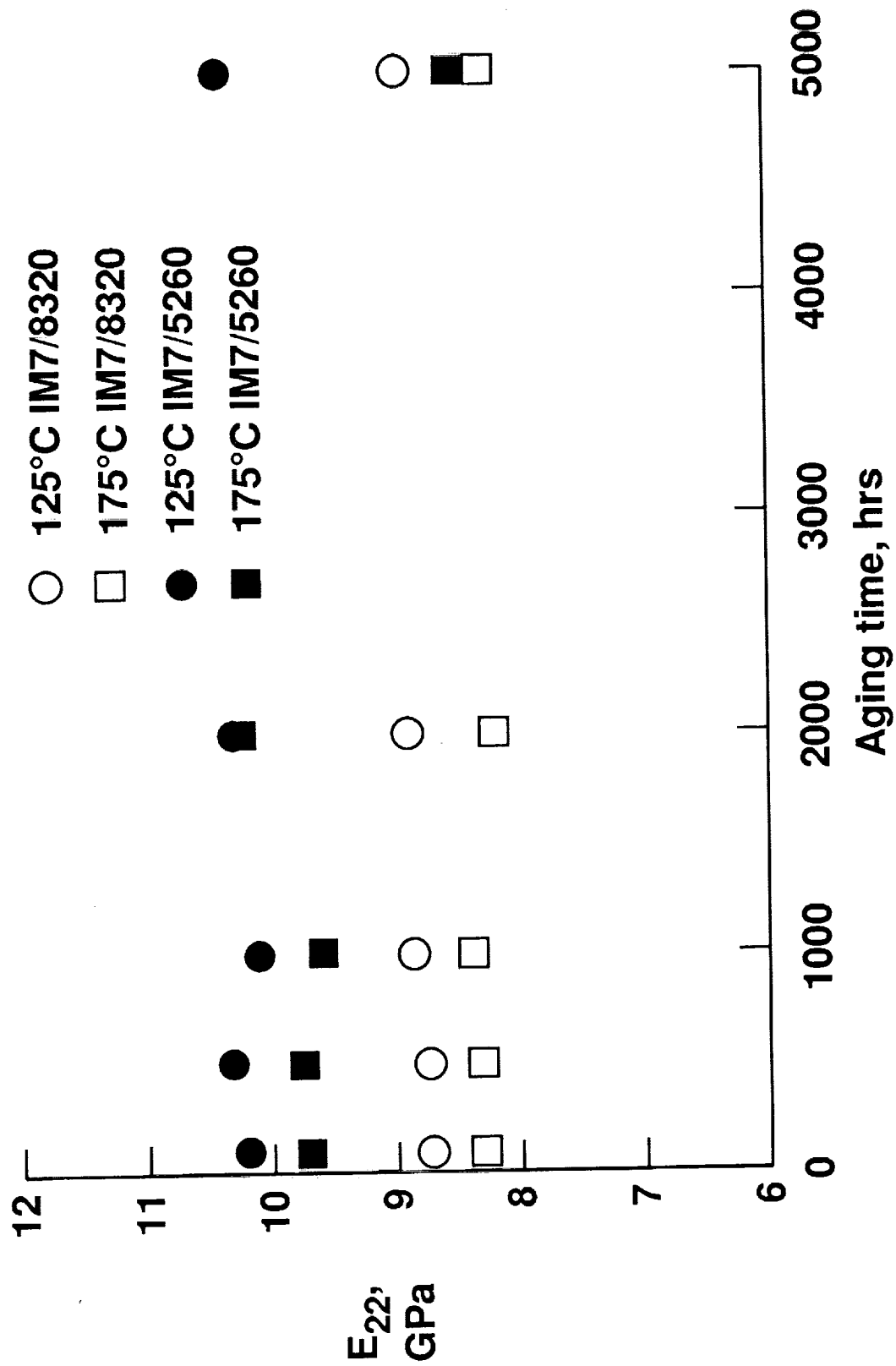


Fig. 7 Transverse modulus (E_{22}) versus aging time measured at room temperature

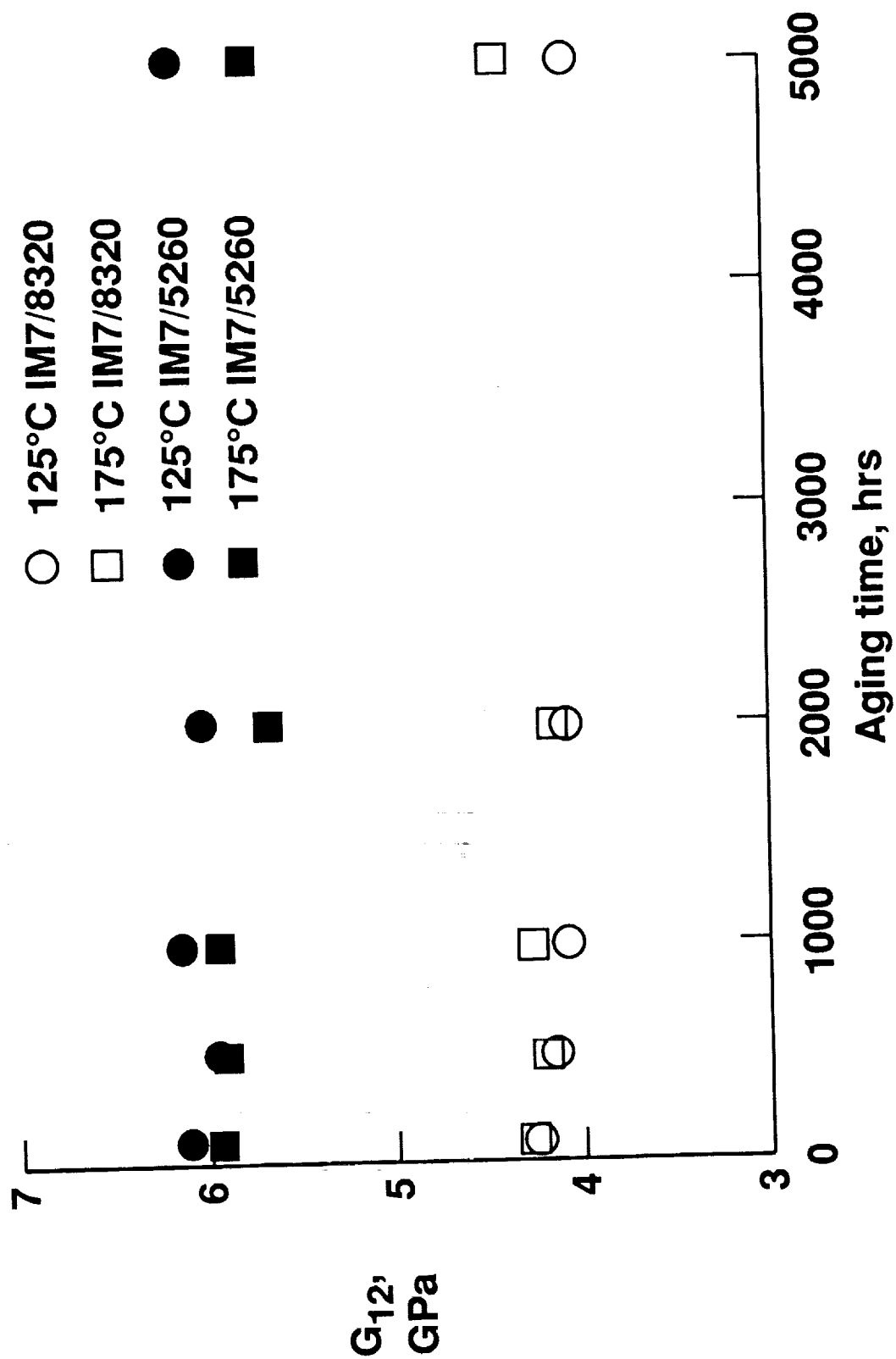


Fig. 8 In-plane shear modulus (G_{12}) versus aging time measured at room temperature

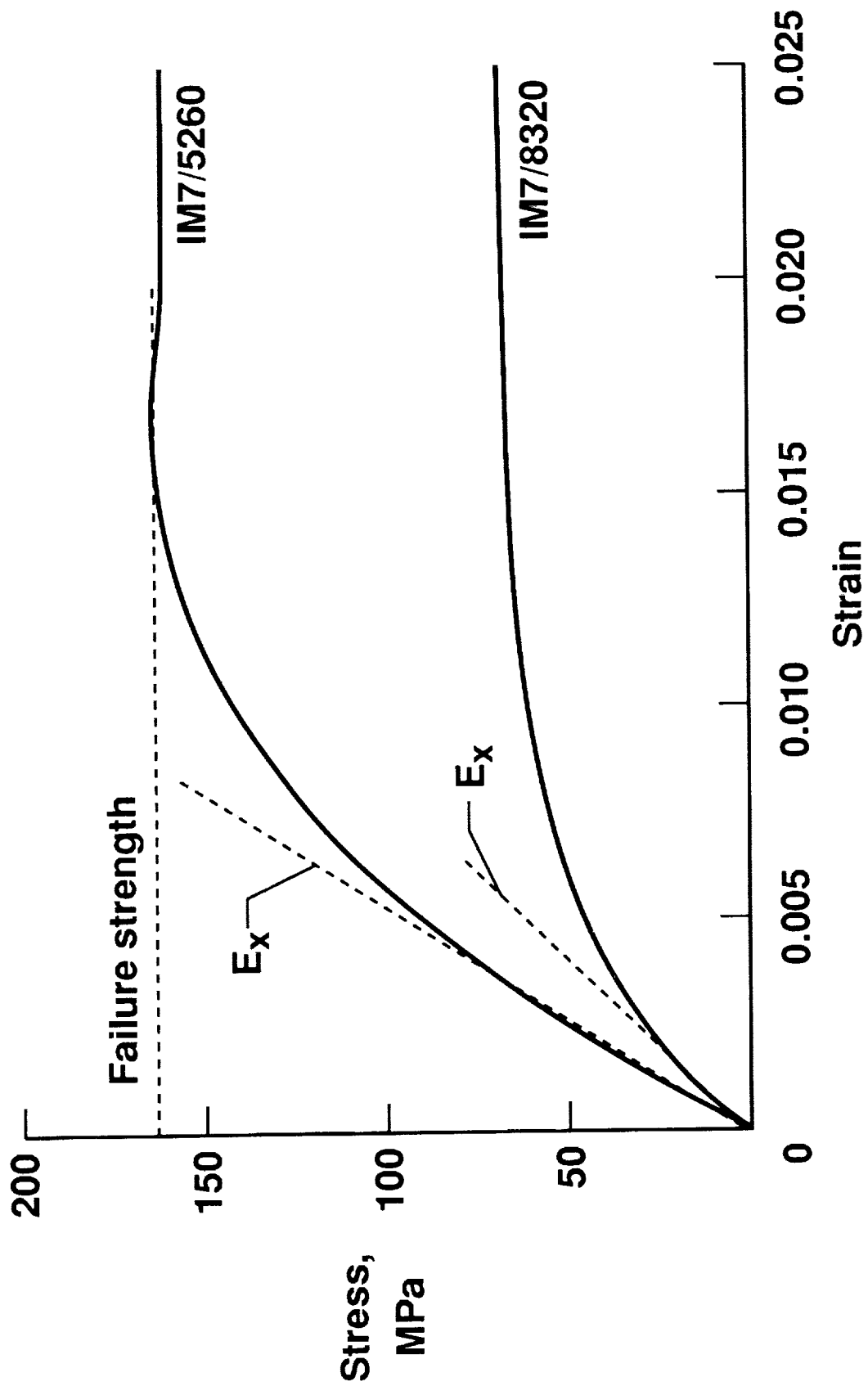


Fig. 9 Longitudinal stress strain curves at room temperature for a $[\pm 45]_{2s}$ laminate after 2000 hours of aging at 175°C

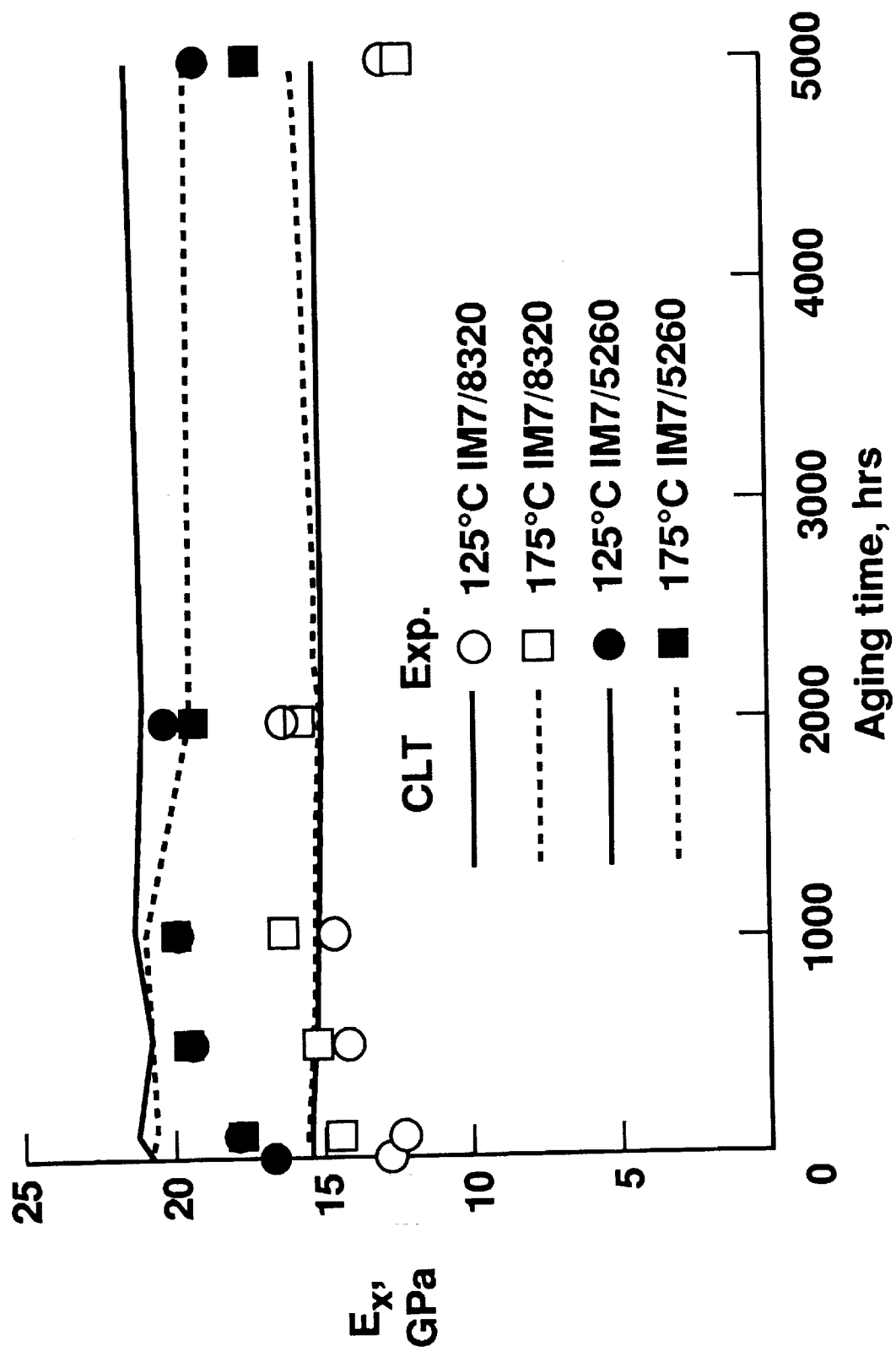


Fig. 10 Longitudinal modulus ($E_{x'}$) of a $[\pm 45]_{2s}$ laminate versus aging time measured at room temperature

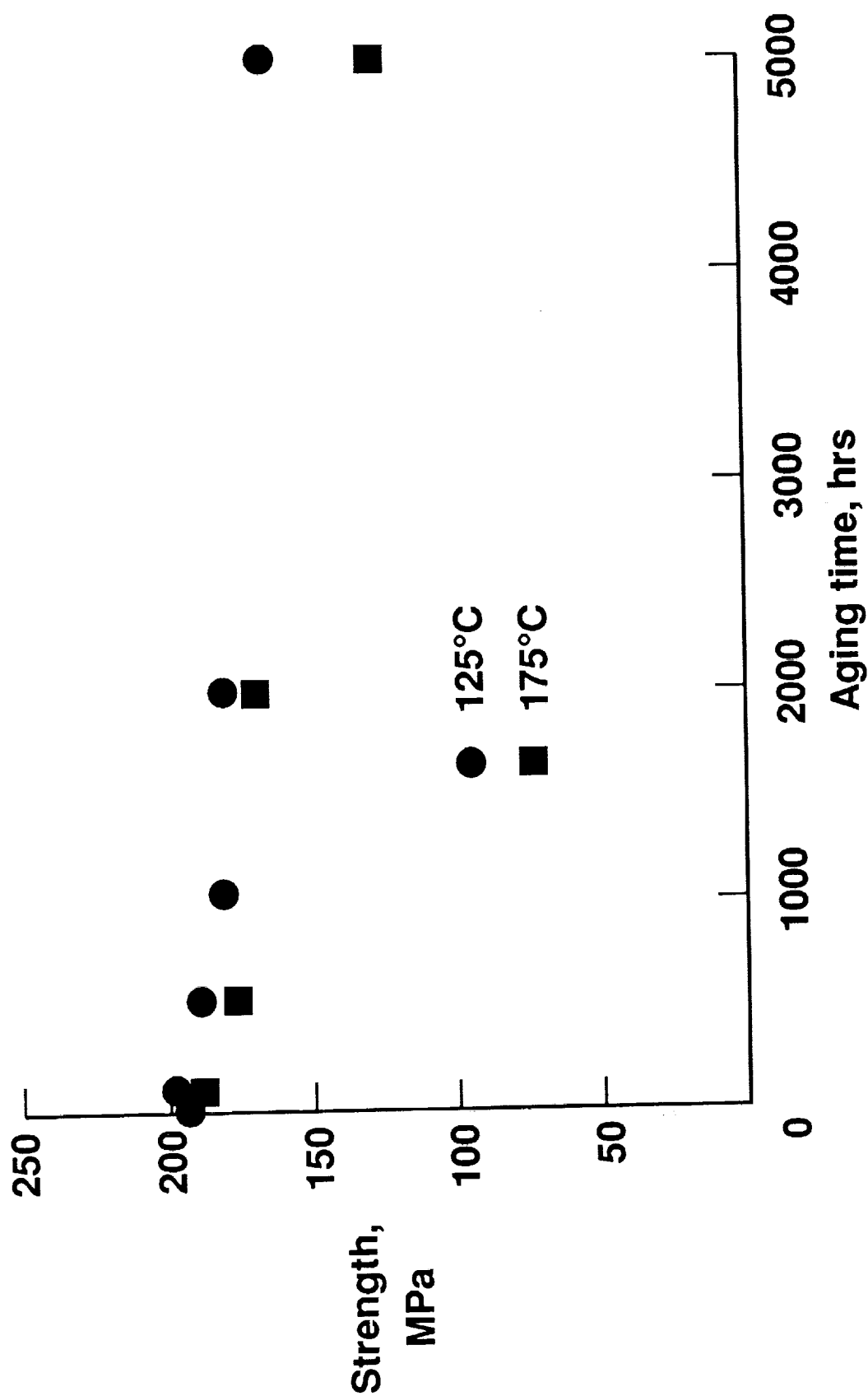


Fig. 11 Longitudinal strength of a $[\pm 45]_{2s}$ IM7/5260 laminate versus aging time measured at room temperature

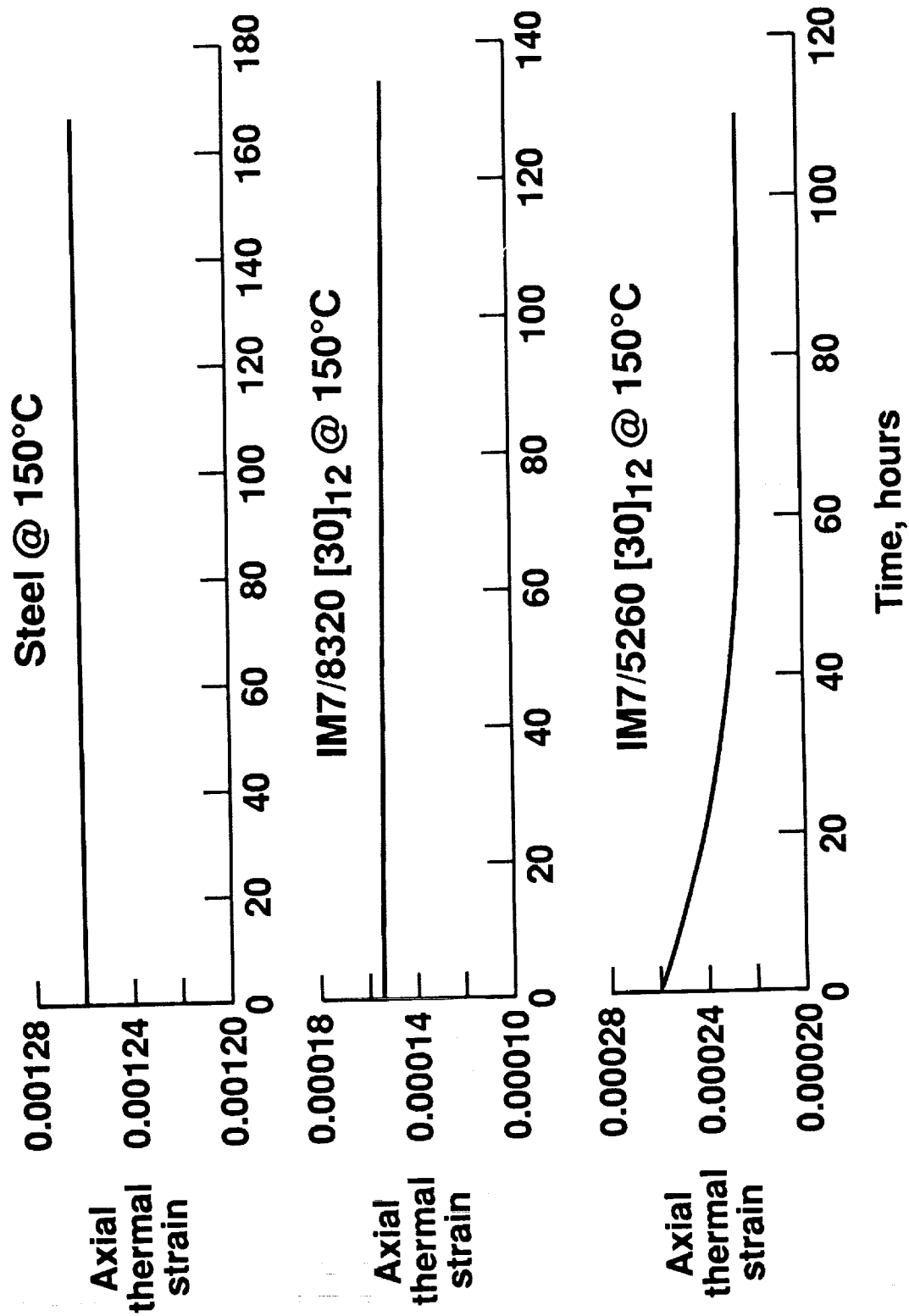


Fig. 12 Free thermal strain variation with time for three different materials

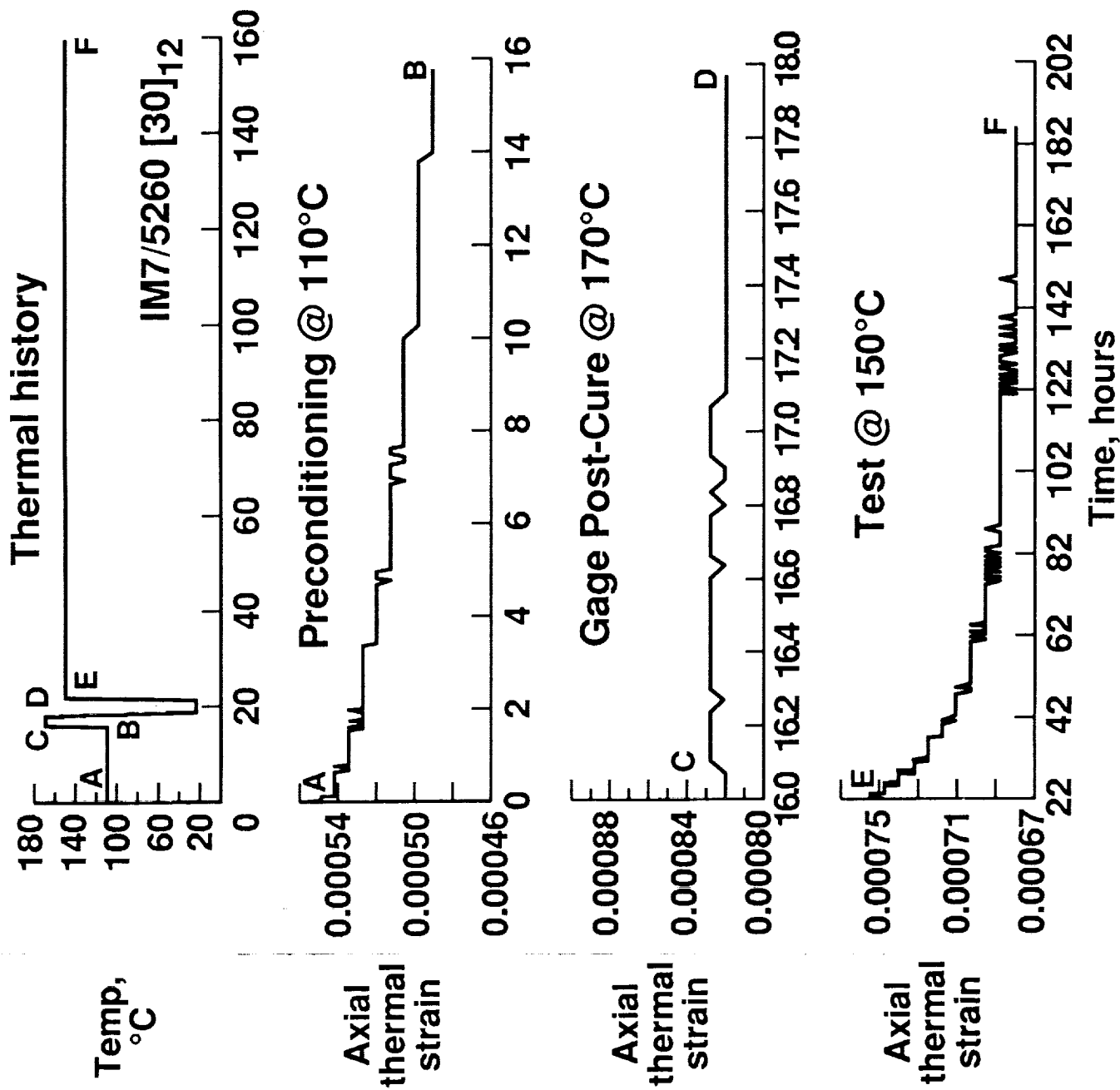


Fig. 13 Free thermal strain variation with time for IM7/5260 specimen

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13. ABSTRACT (Maximum 200 words) Isothermal aging was conducted on two composite systems being considered as possible candidates for the next generation supersonic transport. The composite systems were IM7/5260, carbon/thermoset (toughened bismaleimide) and IM7/8320, carbon/amorphous thermoplastic. The materials were isothermally aged for a total of 5000 hours at 125°C and 175°C. These temperatures are approximately equivalent to the upper skin temperatures of an aircraft flying at Mach 2.0 and Mach 2.4 flight, respectively. The variations of the following properties were determined as a function of aging time: weight loss, moduli, glass transition temperature, microcracking, and modulus and strength of a ± 45 laminate. The difficulties and accuracy of strain measurements are also discussed.				
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